Validating Chlorinated Herbicides
GC, SW-846, Method 8151A

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Annual Review

Reviewed by:_____ Date:_____

Reviewed by:_____ Date:_____

1.0 Traffic Reports and Laboratory Narrative

1.1 Are Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated (J). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced (4°C) upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Has the technical holding times,determined from date of sample receipt to date of extraction, been exceeded? ___ [ ] ___

Note: Samples may be analyzed for herbicide ester and acid. Check Laboratory SDG Narrative.

Note: Aqueous samples must be extracted within 7 days. Extracts must be analyzed within 40 days following extraction.
Soil/Concentrated Waste samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.

ACTION: If technical holding times are exceeded, flag all positive results and non-detects(U)as estimated ("J") and document in the narrative that holding times were exceeded.
Samples extracted more than 28 days from sample receipt, either on the first analysis or upon re-analysis, flag all positive results as

YES NO N/A

estimate ("J") and non-detects as unusable (R).

3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Are the Herbicide Surrogate Recovery Summaries (Form II/Equivalent) present for each of the following matrices?

a. Aqueous [ ] ___ ___

b. Soil [ ] ___ ___

3.2 Are all the samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?

a. Aqueous [ ] ___ ___

b. Soil/Concentrated Waste [ ] ___ ___

ACTION: Contact lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with red pencil.

Note: recommend surrogate is 2,4-Dichlorophenylacetic acid (DCAA)

3.4 Did the laboratory provide their developed in-house QC limits/recoveries? [ ] ___ _____

ACTION: If no, use 70 -130% recovery to qualify data

ACTION: No qualification is done if the surrogate is diluted out. If recovery for the surrogate is below the QC limit, but above 10%, flag all results for that sample "J". If recovery is < 10%, qualify postive results "J" and flag non-detects "R". If recovery is above the QC limits limit, qualify positive values "J".

Note: In-house QC limits must be examined for reasonableness, e.g. 10-170% may be appropriate for analytes not present in the sample.

YES NO N/A

Note: Matrix effect is indicated if the LCS data are within limits but surrogate data exceeds QC limits.

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point calibration analysis? [ ] ___ ___

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgement.

3.6 Are there any transcription/calculation errors between raw data and Form II/Equivalent? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III/Equivalent) present? [ ] ___ ___

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices?

Note: At a minimum, analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair with each batch of up to 20 samples.

a. Aqueous [ ] ___ ___

b. Soil/Concentrated Waste [ ] ___ ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 Did the laboratory provide their developed in-house QC limits/recoveries? [ ] ___ ___

ACTION: If no, use 70 -130% recovery to qualify data

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike results in conjunction with

YES NO N/A

other QC criteria (e.g. LCS) to determine the need for qualification of the data.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis: has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take the action specified above in 3.2. If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 Has a Herbicide instrument blank been analyzed at the beginning of every analytical sequence of 10 samples ? [ ] ___ ___

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

5.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Herbicides? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

YES NO N/A

6.1 Do any method/instrument/reagent/cleanup blanks have positive results for Herbicides? When applied as described in table below, the contaminant concentration in the method blank is multiplied by the sample dilution factor and corrected for % moisture when necessary. ___ [ ] ___

6.2 Do any field/rinse blanks have positive Herbicides results? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, calibration, or any QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 <u>Calibration and GC Performance</u>

7.1 Are the Gas Chromatograms and Data Systems

	YES	NO	N/A
printouts for both columns present for all samples, blanks, QC Check references, and matrix spikes?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

		YES	NO	N/A
7.2	Are Form VI/Equivalent present and complete for each column and each analytical sequence?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

		YES	NO	N/A
7.3	Are there any transcription/calculation errors between raw data and Forms VI?	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

		YES	NO	N/A
7.4	Were the retention time windows calculated using the average absolute retention time (at least three measurements) ± three times the standard deviation of the absolute retention time, for each standard? (Refer to Method 8000A, section 7.5).	[ ]	___	___
7.5.	Was a LCS check standard analyzed prior to environmental samples?	[ ]	___	___
7.5.1	If yes, was the surrogate recovery >50%?	[ ]	___	___
7.5.2	Was the LCS check standard re-extracted/re-analyzed, if surrogate recovery was <50%, or any one analyte was < 40%, or two analytes < 70% ?	[ ]	___	___

Action: If No/' to any of the above, then qualify positive hits as estimated "J" and non-detects as rejected "R" in the original analysis of all samples in the associated analytical sequence.

		YES	NO	N/A
7.6	Do all standard retention times, including each Herbicides in each level of Initial Calibration fall within the windows established during the initial calibration analytical sequence? (For Initial Calibration Standards, Form VI/Equivalent - Herbicides - 1).	[ ]	___	___

ACTION: If no, all samples in the entire analytical sequence are potentially

YES NO N/A

affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogate is visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.7 Are the linearity criteria for the Initial Calibration analyses within limits for both columns? (% RSD must be < 20.0% for all analytes). [ ] ___ ___

ACTION: If no, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When RSD >90%, flag all non-detect results for that analyte R (unusable).

7.8 Are there any transcription/calculation errors between raw data and Form VII - Herbicides-2? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

7.9 Is the resolution between any two adjacent peaks in the QC Reference Check Mixture > 60.0% for both columns? (Form VI-Herbicides- 4) [ ] ___ ___

ACTION: If no, positive results for compounds that were not adequately resolved should be qualified "J". Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable (R).

7.10 Is Form VII -Continuing Calibration present and complete for each analytical sequence for both columns? [ ] ___ ___

ACTION: If no, take action as specified in

3.2 above.

7.11 Have all samples been injected within a 24 hr. period beginning with the injection of the first standard? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly.

7.12 Do all analyte retention times for the Mid-concentration Check standard (Form VII Herb-2) fall within the windows established by the initial calibration sequence? [ ] ___ ___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present

and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.13 Are RPD values for all verification calibration standard compounds < 25.0% [ ] ___ ___

ACTION: The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria.

If %D is 25 -50% qualify as "J"
If %D is 51-100% qualify as "NJ"
If %D is >100% qualify as "R"
If %D is >100% with visible interferences/qualify as "JN"

8.0 Analytical Sequence Check (Form VIII)

8.1 Is Form VIII present and complete for each column

	YES	NO	N/A
and each period of analyses?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

	YES	NO	N/A
8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? (see SAS Client Request/section 8/paragraph 6)	[ ]	___	___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

9.0 Herbicides Identification

	YES	NO	N/A
9.1 Is Form X complete for every sample in which a Herbicide was detected?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

	YES	NO	N/A
9.2 Are there any transcription/calculation errors between raw data and Form X.	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors in data assessment.

	YES	NO	N/A
9.3 Are retention times (RT) of sample compounds within the established RT windows for both columns?	[ ]	___	___
Was GC/MS confirmation provided instead of confirmation by a second dissimilar column?	[ ]	___	___

Action: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis or by GC/MS. Also qualify as unusable (R) all positive results not meeting RT window unless associated standard compounds show a similar RT shift. The reviewer should use professional judgement to assign an appropriate quantitation limit.

9.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns

	YES	NO	N/A
< 25.0%?	[ ]	___	___

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
25-50 %	J
50-90 %	JN
> 90 %	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

9.5 Check chromatograms for false negatives.
Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values.
Were any errors found? ___ [ ] ___

NOTE: The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity (NJ). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate the presence of interferences during the evaluation of the second column confirmation.

YES NO N/A

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

10.3 Have all data (Forms and associated chromatograms and quantitation reports) been submitted for original, diluted or re-extraction/re-analysis samples? [ ] ___ ___

11.0 Chromatogram Quality

11.1 Were baselines stable? [ ] ___ ___

11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

ACTION: Address comments under System Performance of data assessment. Explain use of professional judgement where used to qualify data.

YES NO N/A

12.0 Field Duplicates

12.1 Were any field duplicates submitted for Herbicides analysis? [ ] ___ ___

Note: Check whether SAS Client Request required field duplicates.

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.